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Crystal data (II) for some androstanes*.

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Nort n, Lu & Campbell (1962) reported the single-crystal data for a series of androstanes. This paper reports the same data for a second set of androstanes (Table 1) using the same techniques described in the earlier paper. No further work on these compounds is contemplated.

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Reference

NORTON, D. A., LU, C. T. & CAMPBELL, A. E. (1962). *Acta Cryst.* **15**, 1189.

Table 1. *Crystal data* (II) *for some androstanes*

	1	2	3	4	5	6	7	8	9
Formula	$C_{19}H_{26}O_2$	$C_{19}H_{26}O_2$	$C_{19}H_{28}O_2$	$C_{19}H_{28}O_2$	$C_{19}H_{30}O_2$	$C_{19}H_{30}O_2$	$C_{19}H_{24}O_3$	$C_{21}H_{30}O_3$	$C_{26}H_{34}O_3$
Mol. Wt.	286·40	286·40	288·41	288·41	290·43	290·43	300·38	330·45	394·53
D_m (g.cm^{-3})	1·178	1·164	1·147	1·186	1·132	1·147	1·264	1·162	1·148
D_x (g.cm^{-3})	1·175	1·174	1·143	1·177	1·103	1·084	1·253	1·201	1·207
Space group	$P2_12_12_1$	$P2_12_12$	$A2$	$P2_1$	$P2_1$	$P2_12_12_1$	$P2_12_12_1$	$P2_12_12_1$	$P2_1$
Z	4	4	4	4	2	4	4	4	2
a (Å)*	12·963	12·302	21·337	14·691	11·614	12·146	9·263	12·800	10·860
b (Å)*	16·929	18·644	6·186	11·093	8·096	23·434	26·531	18·169	16·241
c (Å)*	7·366	7·065	12·704	10·872	9·422	6·248	6·477	7·856	6·236
β (°)	—	—	91·27	113·23	99·23	—	—	—	99·17
V (Å^3)	1619	1620	1676	1628	874	1779	1592	1827	1086
Solvent	Ethanol	Methanol	Unknown	Methanol	Ethanol	Ethanol	Methanol—acetone	Methanol	Ethanol

* ± 0·009 Å

1. 4-Androsten-3,17-dione (androstenedione)
2. 1,4-Androstadien-17β-ol-3-one (1-dehydrotestosterone)
3. 5α-Androstan-3,17-dione (androstanedione)
4. 4-Androsten-17β-ol-3-one (testosterone)
5. 5α-Androstan-17β-ol-3-one (allodihydrotestosterone)
6. 5-Androsten-3β, 17β-diol (adrenostenediol)
7. 4-Androsten-3,11,17-trione (adrenosterone)
8. 4-Androsten-17β-ol-3-one 17-acetate (testosterone acetate)
9. 5α-Androstan-17β-ol-3-one 17-benzoate (dihydrotestosterone benzoate)

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An X-ray investigation of the stereochemistry of $Zn(NCS)_2(C_6H_5NH_2)_2$.

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The stereochemistry of the complex $Zn^{II}(NCS)_2(C_6H_5NH_2)_2$ is of interest in connection with studies in these laboratories on the factors governing the configuration of metal complexes. Nelson & Shepherd (unpublished work) have established, from magnetic and spectral data, that the corresponding Co(II) and Ni(II) complexes have octahedral (or tetragonal) structures with bridging NCS groups. X-ray powder patterns show that these and the corresponding Cd(II) complex are isomorphous, but that $Zn(NCS)_2(C_6H_5NH_2)_2$ has a different crystal structure. An attempt to determin this structure sufficiently to establish the coordination number of the zinc atom is described here.

Single crystals obtained by recrystallization from ethanol were used. These were needle-shaped, approx 1·5 mm long, and 0·3 mm in cross section. The needle axis being taken as the c axis, rotation photographs with Cu $K\alpha$ radiation about the [001] and [1$\bar{1}$0] axes and Laue photographs showed the unit cell to be orthorhombic with the cell dimensions:

$$a = 14{\cdot}56 \pm 0{\cdot}05 \text{ Å}$$
$$b = 9{\cdot}10 \pm 0{\cdot}05$$
$$c = 12{\cdot}7 \pm 0{\cdot}1$$

The values f a and b were derived from the observed spacings of 39 $h0l$ reflexions, and their uncertainties are an estimate based on the standard deviations of the observed and calculated values. c was obtained from layer line measurements of a rotation photograph.

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Crystallographic data for testoster ne hydrate and anhydrate.

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Testosterone hydrate crystallizes in the space group $P2_12_12_1$ with four molecules in a unit cell having the dimensions $a=13{\cdot}63$, $b=15{\cdot}95$ and $c=7{\cdot}94$ Å. Anhydrous testosterone crystallizes in the space group $P2_1$ with four molecules in the unit cell. The proper cell dimensions are $a=14{\cdot}45$, $b=11{\cdot}09$, $c=10{\cdot}88$ Å and $\beta=110{\cdot}5°$.

In a previous study on the solution behavior of testosterone in aqueous media, conversion of the anhydrate form to a hydrate was reported (Thakkar & Hall, 1969). Since testosterone is a natural hormone and exists in an aqueous environment, characterization of this form is important. We wish to report here the crystallographic parameters of the hydrate.

Small single-crystals were grown by a continuous fall method from saturated aqueous solution cooled from 33·0 to 29·5°C at 0·1°C per hour. Elemental analysis, Karl Fischer titration and thermogravimetric analysis showed this crystalline form to be the monohydrate.

From Weissenberg and precession photographs taken with Cu $K\alpha$ radiation the space group has been found to be $P2_12_12_1$ (systematic absences: $h00$, $0k0$, and $00l$ for h, k or l odd); there are four molecules in a unit-cell having the dimensions $a=13{\cdot}63$, $b=15{\cdot}95$ and $c=7{\cdot}94$ Å. The density measured by displacement is 1·181 g.cm^{-3}, which agrees well with the calculated density for $C_{19}H_{28}O_2.H_2O$ of 1·179 g.cm^{-3}.

For comparison we have measured the crystal parameters for anhydrous testosterone and have obtained values which differ from those reported by Bernal & Crowfoot (1936). The space group is $P2_1$ with four molecules in a unit-cell having the dimensions $a=14{\cdot}73$, $b=11{\cdot}09$, $c=10{\cdot}88$ Å and $\beta=113{\cdot}3°$, which agree fairly well with the values given by Ohrt, Haner & Norton (1965). There is, however, an alternative cell with β closer to 90°. The dimensions for this proper cell are $a=14{\cdot}45$, $b=11{\cdot}09$, $c=10{\cdot}88$ Å and $\beta=110{\cdot}5°$. These cells give a calculated density of 1·173 g.cm^{-3}, which is identical with the experimentally measured value given by Bernal & Crowfoot.

The indexed powder data for these two forms of testosterone will be submitted for inclusion in the *ASTM Powder Diffraction File*.

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Crystal data of $BaSrFe_4O_8$.

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The dimensions of the orthorhombic unit cell of $BaSrFe_4O_8$, which contains two formula units, are $a=5{\cdot}516$, $b=8{\cdot}265$, $c=9{\cdot}188$ Å. The sapce group is *Pnna*.

A previous report on the phase equilibrium diagram, BaO–SrO–Fe_2O_3, shows that a new stable compound, having the composition $BaSrFe_4O_8$, may occur as a single phase above 1100 ± 10°C (Batti, 1962). It undergoes thermal transformation at about 1200°C and melts incongruently at 1240 ± 10°C. A further investigation by Barbariol & Batti (1968) established that this new phase forms a solid solution with the binary compound $BaFe_2O_4$, which is reported to be orth rhombic (Okazaki, Mori & Mitsuda, 1963; DoDinh & Bertaut, 1965). They display complete solubility above 1200°C whereas at lower temperatures a solid-solution gap of increasing width was reported.

Single crystals of $BaSrFe_4O_8$ were gr wn, by solid-state reacti n, from a pressed pellet mixture f 1 $BaCO_3$: 1 $SrCO_3$: 2 Fe_2O_3 which was heated on a platinum strip in a resistance furnace to about 950°C. The sintered pellet was reground and refired to assure complete reaction. The microcrystalline specimen was br ught to 1200°C and left in the furnace

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